

PATENT ABSTRACTS OF JAPAN

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(54) CORROSION RESISTANT MEMBER

(57)Abstract:

PROBLEM TO BE SOLVED: To solve the problem of unstable characteristics of a compd. of groups IIa and IIIa elements of the Periodic Table having high corrosion resistance while a sintered compact of glass, quartz, stainless steel, alumina or AlN used so far has unsatisfactory corrosion resistance.

SOLUTION: A part of a member exposed to halogen-contg. corrosive gas such as fluorine- or chlorine-contg. gas or plasma of the gas is made of a ceramic sintered compact based on a compd. contg. at least one of groups IIa and IIIa elements of the Periodic Table, e.g. a multiple oxide, spinel, cordierite, YAG or a silicate and having $\leq 4 \mu\text{m}$ surface roughness Ra and $\leq 3\%$ porosity.

CLAIMS

[Claim(s)]

[Claim 1] the part put to halogen nature corrosion gas or those plasma, such as a fluorine system and a chlorine system, -- the [periodic table] -- the [2a group and] -- the corrosion-resistant member which makes a subject the compound containing at

least one sort in 3a group element, and is characterized by for the surface roughness (Ra) being constituted by 1 micrometer or less, and constituting porosity with 3% or less of ceramic sintered compact.

DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the corrosion-resistant member used as fixtures, such as wall material which has high corrosion resistance to halogen system corrosive gas and the halogen system plasma especially, and which is used inside plasma treatment equipment, or the object for semi-conductor manufacture or the plasma equipment for liquid crystal processes, wafer support, a protective cover, and an insulating ring.

[0002]

[Description of the Prior Art] Utilization of plasma, such as a dry process of semi-conductor manufacture, plasma jet flame coating, the discharge tube, and a plasma display, is progressing quickly in recent years. As a plasma process in a semi-conductor, a fluorine system and the halogen system corrosion gas of ***** are used for vapor growth, etching, or cleaning from the reactant height.

[0003] Corrosion resistance with the high member in contact with these corrosive gas and plasma is required.

[0004] Generally the members which contact these plasma other than a processed material conventionally are SiO_2 , such as glass and a quartz. Corrosion-resistant metals, such as an ingredient used as a principal component, and stainless steel, Monel, are used abundantly.

[0005] Moreover, it is used noting that what carried out surface coating of an alumina sintered compact, sapphire, the sintered compact of AlN, or these with the CVD method etc. by making a wafer into the susceptor material which carries out support immobilization at the time of semi-conductor manufacture manufacture is excellent in corrosion resistance. Moreover, the heater which coated graphite and boron nitride is used.

[0006]

[Problem(s) to be Solved by the Invention] however, if glass and the quartz of the corrosion resistance in the plasma which are used from the former are inadequate, and consumption is intense and touches especially the halogen plasma, the contact surface will etch -- having -- a front face -- description changed, and in the member for which light transmission nature is needed, a front face is white gradually and the problem of translucency falling had produced the overcast. Moreover, since the corrosion resistance of the member which used metals, such as stainless steel, is inadequate, especially in semi-conductor manufacture, it becomes the cause of defective generating by corrosion.

[0007] Although an alumina and the sintered compact of AlN are excellent in corrosion resistance to halogen system gas as compared with the above-mentioned ingredient, when the plasma was touched at the elevated temperature, corrosion advanced gradually, degaining of a crystal grain child arose from the front face of a sintered compact, and the problem of becoming the cause of particle generating has occurred.

[0008] If a reaction with halogen system corrosion gas or the plasma advances first as

a result of repeating an examination of an ingredient excellent in the corrosion resistance over halogen system corrosion gas and the plasma, this invention persons the [that a halogenide is generated by the front face and / periodic table] -- the [2a group and] -- the [periodic table / since the melting point is high and the halogenide containing 3a group element is stable] -- the [2a group and] -- it proposed previously that 3a group compound was suitable as a corrosion-resistant member. [0009] Also in it, since it has moderate reinforcement while it is applicable to all configuration articles, and these sintered compacts are applicable also as the various structures, the usefulness is high. the [however, / periodic table] -- the [2a group and] -- in the sintered compact containing 3a group element, there was a problem that corrosion resistance was not stabilized and there was a problem that corrosion resistance changed with the organizations and surface states of a sintered compact.

[0010]

[Means for Solving the Problem] Then, as a result of repeating examination, even if this invention persons are periodic table the 2a excellent in corrosion resistance, and 3a group element compound If the front face put to the plasma of an ingredient is coarse, the area which contacts corrosive gas and the plasma so much will become large, and corrosion resistance will fall, When pore exists in a sintered compact, progress of corrosion in the pore part which appeared in the front face Moreover, early, Since degradation of a surface state is promoted and the life of an ingredient was shortened when halogen gas was contacted, the knowledge of the ability to control the effect was carried out by controlling the surface roughness and the porosity of a sintered compact on predetermined level.

[0011] namely, the part where the corrosion-resistant member of this invention is put to halogen nature corrosion gas or those plasma, such as a fluorine system and a chlorine system, -- the [periodic table] -- the [2a group and] -- the compound containing at least one sort in 3a group element is made into a subject, and it is characterized by for the surface roughness (Ra) constituting by 1 micrometer or less, and porosity constituting with 3% or less of sintered compact.

[0012] That is, according to research of this invention persons, that the origin of corrosion progress comes on a member front face, if an edge is formed of a blemish, a micro crack, etc. of not only pore but a front face, electric field will concentrate there and progress of corrosion will be accelerated. Also in order to prevent it, it is necessary to finish the front face of a member smoothly. If surface roughness Ra becomes large, concavo-convex formation is promoted by contact to corrosive gas or the plasma, and the life of an ingredient will be reduced by it. For the reason, by setting surface roughness Ra to 1 micrometer or less, concentration of electric field can be prevented and progress of corrosion can be controlled.

[0013] moreover, the front face according to the corrosion of the pore section by making the porosity in a sintered compact into 3% or less similarly -- it becomes possible to prevent the corrosion resistance lowering accompanying degradation of description, and the increment in surface area.

[0014]

[Embodiment of the Invention] The corrosion-resistant member of this invention is a member put to halogen system gas or the plasma. As halogen system gas Fluorine system gas, such as SF₆, NF₃, CF₄, CHF₃, ClF₃, and HF, Cl₂, BCl₃, and SiCl₄ etc. - - chlorine-based gas, and HBr and Br₂ etc. -- it is iodine system gas, such as bromine system gas and HI, etc., and these gas will be plasma-ized if microwave, a RF, etc. are introduced into the ambient atmosphere into which these gas was introduced.

[0015] Moreover, since the etching effectiveness is heightened more, with the gas of a

halogen system, inert gas, such as Ar, is introduced and the plasma may be generated. [0016] the member used in such halogen system corrosion gas or its plasma in the corrosion-resistant member of this invention -- the [periodic table] -- the [2a group and] -- the compound containing at least one sort in 3a group element consists of sintered compacts made into a subject. the [the periodic table 2a group this sintered compact of whose is a configuration element, and] -- in order that 3a group element may react with a halogen and may form a stable compound, it has the corrosion resistance which was markedly alike and was conventionally superior to the ingredient in itself.

[0017] the [which is used / periodic table] -- as 2a group element -- Mg, calcium, and Ba -- suitable -- the [periodic table] -- as a 3a group element, Sc, Y, La, Ce, Nd, Yb, Dy, and Lu are suitable, and an oxide, a nitride, carbide, boride, a fluoride, etc. are mentioned as a compound. These will form the halogenide containing each element by carrying out long duration contact with halogenation gas and those plasma. the [in addition, / said / periodic table] -- the [2a group and] -- as a compound containing 3a group element You may be a multiple oxide containing elements, such as 3b group element, and Si, Pb, Fe, Cr, Ti. the [these elements and /, such as aluminum, / periodic table] -- specifically The spinel mold crystal or cordierite crystal expressed with AB_2O_4 (A the [periodic table] 2a group element and B the [periodic table] 3b group element), the [periodic-table] -- as the compound of 3a group element and aluminum -- a perovskite mold (YAP mold), a melilite mold (YAM mold), and a garnet mold (YAG mold) compound -- the silicate compound of periodic-table the 2a and 3a group element etc. can be further used as a conjugated compound with Si.

[0018] Furthermore, in the sintered compact which constitutes this member, it is important that that surface roughness (Ra) sets 1 micrometer or less to 0.1 micrometers or less more preferably. If the surface roughness of a member exceeds 1 micrometer, the area in contact with corrosive gas or the plasma will become large, and corrosion resistance will fall greatly. Therefore, as for a member front face, it is desirable that mirror plane processing is carried out infinite. Moreover, that the origin of corrosion progress comes in a material-list side, if an edge is formed of a blemish, a micro crack, etc. of not only pore but a front face, electric field will concentrate there and progress of corrosion will be accelerated by them. Also in order to prevent it, it is necessary to finish the front face of a member smoothly.

[0019] Moreover, progress of the local corrosion on the front face of a member and degradation of a surface state can be prevented by making especially into 1% or less the porosity of the sintered compact which constitutes a member 3% or less. Even if it is difficult to acquire a desirable surface state (mirror plane) which was mentioned above if porosity exceeds 3% and it can acquire a mirror plane condition, while being put to the corrosion ambient atmosphere, corrosion will advance from the pore section which remained in the front face, a surface state will deteriorate, and life lowering of a member will be caused.

[0020] As long as the precise sintered compact which fulfills predetermined porosity and the conditions of surface roughness is obtained as a manufacturing method of the corrosion-resistant member of this invention, what kind of approach may be used. For example, with the sintered compact fabricated and calcinated by the well-known approach, a reaction-sintering object, and a well-known sol gel process, the liquid phase may be applied to a predetermined base front face, and may be calcinated.

[0021] concrete -- as a start raw material -- the [periodic table] -- the [3a group element and] -- the metal powder of 2a group element -- Or compound powder, such as an oxide, carbide, a nitride, a carbonate, and acetate, is used. The mixed powder

which carried out weighing capacity so that it might become the same presentation as the sintered compact produced eventually, Or the conjugated compound powder which carried out temporary quenching treatment, ground, and was produced after mixing said start raw material by the predetermined ratio, There is a ceramic precursor of mixed powder with the metal powder which can form a sintered compact by reaction sintering, said compound, or a conjugated compound, the organic system decomposed into the ceramics by heat-treatment, and an inorganic system etc. Moreover, the assistant for promoting sintering if needed can also be added.

[0022] Such a raw material is applied and calcinated in a desired configuration at shaping or a base. As the shaping approach, the shaping approach suitable for acquiring the object configuration, such as the usual dry type press, a hydrostatic-pressure press, a slip casting, extrusion molding, and sheet-like shaping, can be used.

[0023] A Plastic solid is calcinated at the ambient atmosphere which was suitable for eburnation according to the ingredient, a pressure, and temperature, and if required, it should just disappear the pore in a sintered compact by the hydrostatic-pressure method between heat heat-treated in the inert gas of 1000 to 2000 atmospheric pressure.

[0024] Moreover, although a dry area etc. may occur on the sintered compact front face after baking depending on the construction material to be used, in such a case, the corrosion-resistant member of this invention is producible by carrying out mirror-polishing processing to 1 micrometer or less of surface roughness by polish processing of common knowledge of the front face of a sintered compact which contacts gas or the plasma at least.

[0025]

[Example]

The high grade impalpable powder of each ingredient as shown in example 1 table 1 was fabricated, it calcinated at 1300-1800 degrees C, and the sintered compact with which porosity differs was produced. In addition, as for the sample of 0% of porosity, all carry out hydrostatic-pressure baking (HIP) between heat of the sintered compact in the argon gas of 2000 atmospheric pressures. And the sample from which these porosity differs was ground to suitable field granularity with surface grinding and a lap, and each sample from which porosity and field granularity differ was prepared.

[0026] With a RIE plasma etching system, it is SF₆ about these. It put to the plasma at the room temperature for 3 hours, and the etch rate was computed from weight change. About the construction material article same about these etch rates, the etch rate of the other sample when setting the etch rate of a sample with smallest surface roughness Ra and porosity to 1.00 was computed as a relative value, and was shown in a table 1.

[0027]

[A table 1]

[0028] Although the absolute value of an etch rate changes with construction material, respectively, any sample is known by that it will be twice [more than] the sample in which the etch rate carried out HIP processing / mirror plane processing about it if porosity exceeds 3% by surface roughness Ra exceeding 1 micrometer. Therefore, even if it is the same construction material, it is understood that surface roughness and porosity are the big factor which determines corrosion resistance.

[0029] Each sample produced like example 2 example 1 was put to the HCl plasma with the RIE plasma etching system for 3 hours, and the same assessment as an example 1 was performed. The result was shown in a table 2.

[0030]
[A table 2]

[0031] Surface roughness Ra exceeded 1 micrometer like the example 1 also in this case, or when porosity exceeded 3%, the etch rate (relative value) was a thing exceeding 2, so that clearly also from the result of a table 2.

[0032] The sample produced like example 3 example 1 was put to the HBr plasma with the RIE plasma etching system for 3 hours, and the same assessment as an example 1 was performed. The result was shown in a table 2.

[0033]
[A table 3]

[0034] Surface roughness Ra exceeded 1 micrometer like the example 1 also in this case, or when porosity exceeded 3%, the etch rate (relative value) was a thing exceeding 2, so that clearly also from the result of a table 3.

[0035]
[Effect of the Invention] The corrosion-resistant member of this invention by controlling the porosity and surface roughness of a sintered compact which constitute the member put to halogen system corrosive gas and its plasma to specific within the limits as explained in full detail above It becomes possible to fully pull out the original corrosion resistance which the sintered compact ingredient has. By that cause Wall material, a fixture, etc. in a semi-conductor or the plasma treatment equipment for liquid crystal manufacture can attain reinforcement by specifically using it for an insulating ring, electrode covering, etc. of the circumference of the clamp ring for wafer immobilization, and the electrode of an etching system.

TECHNICAL FIELD

[Field of the Invention] This invention relates to the corrosion-resistant member used as fixtures, such as wall material which has high corrosion resistance to halogen system corrosive gas and the halogen system plasma especially, and which is used inside plasma treatment equipment, or the object for semi-conductor manufacture or the plasma equipment for liquid crystal processes, wafer support, a protective cover, and an insulating ring.

PRIOR ART

[Description of the Prior Art] Utilization of plasma, such as a dry process of semi-conductor manufacture, plasma jet flame coating, the discharge tube, and a plasma display, is progressing quickly in recent years. As a plasma process in a semi-conductor, a fluorine system and the halogen system corrosion gas of ***** are used for vapor growth, etching, or cleaning from the reactant height.

[0003] Corrosion resistance with the high member in contact with these corrosive gas and plasma is required.

[0004] Generally the members which contact these plasma other than a processed material conventionally are SiO(s)2, such as glass and a quartz. Corrosion-resistant

metals, such as an ingredient used as a principal component, and stainless steel, Monel, are used abundantly.

[0005] Moreover, it is used noting that what carried out surface coating of an alumina sintered compact, sapphire, the sintered compact of AlN, or these with the CVD method etc. by making a wafer into the susceptor material which carries out support immobilization at the time of semi-conductor manufacture manufacture is excellent in corrosion resistance. Moreover, the heater which coated graphite and boron nitride is used.

EFFECT OF THE INVENTION

[Effect of the Invention] The corrosion-resistant member of this invention by controlling the porosity and surface roughness of a sintered compact which constitute the member put to halogen system corrosive gas and its plasma to specific within the limits as explained in full detail above It becomes possible to fully pull out the original corrosion resistance which the sintered compact ingredient has. By that cause Wall material, a fixture, etc. in a semi-conductor or the plasma treatment equipment for liquid crystal manufacture can attain reinforcement by specifically using it for an insulating ring, electrode covering, etc. of the circumference of the clamp ring for wafer immobilization, and the electrode of an etching system.

TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] however, if glass and the quartz of the corrosion resistance in the plasma which are used from the former are inadequate, and consumption is intense and touches especially the halogen plasma, the contact surface will etch -- having -- a front face -- description changed, and in the member for which light transmission nature is needed, a front face is white gradually and the problem of translucency falling had produced the overcast. Moreover, since the corrosion resistance of the member which used metals, such as stainless steel, is inadequate, especially in semi-conductor manufacture, it becomes the cause of defective generating by corrosion.

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and] -- in the sintered compact containing 3a group element, there was a problem that corrosion resistance was not stabilized and there was a problem that corrosion resistance changed with the organizations and surface states of a sintered compact.

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[Means for Solving the Problem] Then, as a result of repeating examination, even if this invention persons are periodic table the 2a excellent in corrosion resistance, and 3a group element compound If the front face put to the plasma of an ingredient is coarse, the area which contacts corrosive gas and the plasma so much will become large, and corrosion resistance will fall, When pore exists in a sintered compact, progress of corrosion in the pore part which appeared in the front face Moreover, early, Since degradation of a surface state is promoted and the life of an ingredient was shortened when halogen gas was contacted, the knowledge of the ability to control the effect was carried out by controlling the surface roughness and the porosity of a sintered compact on predetermined level.

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EXAMPLE

[Example]

The high grade impalpable powder of each ingredient as shown in example 1 table 1 was fabricated, it calcinated at 1300-1800 degrees C, and the sintered compact with which porosity differs was produced. In addition, as for the sample of 0% of porosity, all carry out hydrostatic-pressure baking (HIP) between heat of the sintered compact in the argon gas of 2000 atmospheric pressures. And the sample from which these porosity differs was ground to suitable field granularity with surface grinding and a lap, and each sample from which porosity and field granularity differ was prepared.

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